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# **Enhancement of Convective Heat Transfer for Electronic Devices**

## **Application using Patterned Multiwall Carbon Nanotubes Structures**

**M. I. Shahzad<sup>1</sup>, M. Giorcelli<sup>1</sup>, L. Ventola<sup>2</sup>, D. Perrone<sup>3</sup>, N. Shahzad<sup>1,3</sup>, E. Chiavazzo<sup>2</sup>,  
P. Asinari<sup>2</sup>, M. Cocuzza<sup>1,4</sup>, A. Tagliaferro<sup>1</sup>**

<sup>1</sup>Department of Applied Science and Technology, Politecnico di Torino, Corso Duca degli  
Abruzzi 24, 10129 Torino, Italy

<sup>2</sup>SMaLL - Multi Scale Modeling Laboratory, Energy Department, Politecnico di Torino, 10129  
Torino, Italy

<sup>3</sup>Center for Space Human Robotics, Italian Institute of Technology, Corso Trento 21, 10129  
Torino, Italy

<sup>4</sup>CNR-IMEM, Parco Area delle Scienze 37/A, 43124 Parma, Italy

***Corresponding Author:*** Muhammad Imran Shahzad  
Email: imran.shahzad@polito.it, Ph: +39 011 0907380, Fax: +390110907399  
Department of Applied Science and Technology (DISAT),  
Politecnico di Torino, Corso Duca degli Abruzzi 24, 10129 Torino, Italy.

## **ABSTRACT**

*This article reports on the heat transfer characteristics of columnar Vertically Aligned Multiwall Carbon Nanotubes (VA-MWCNTs) grown on a patterned Si surface. In the first part, we describe the procedure for patterning the silicon surface so that the growth of predetermined MWCNTs structures is obtained. The dimensions of CNT structures are in the range of few hundred micrometers while the diameter of single MWCNT was in the range of 30-80 nm. In the second part structures mimicking macroscopic finned heat sinks are used for enhancing forced convective heat transfer on a silicon substrate. Convective heat transfer coefficient has been experimentally measured for silicon substrates with and without MWCNT-based fins on it. The configuration with MWCNTs shows an enhancement in convective heat transfer of 40% and 20%, as maximum and average value respectively, compared to the bare silicon. Experiments have been carried out in a wind tunnel with air as coolant in fully turbulent regime. These encouraging results and the possibility of growing structures directly on silicon can be regarded as a first step towards chip cooling applications based on MWCNT structures.*

## ***INTRODUCTION***

The publication on the characterization of carbon nanotubes (CNTs) by Iijima in 1991 [1] helped to ignite a still ongoing research in carbon-based materials. CNTs have exceptional physical, electrical, thermal and mechanical properties and have opened up a new subject in materials sciences [2]. These materials have been excellent candidate for many applications in the field of nanoelectronic technology and super-strong reinforced composite material [3-5]. CNTs can be grown by various processes [6] including arc discharge, laser ablation and Chemical Vapor Deposition (CVD). The most promising of these techniques is CVD because of the possibility of large scale production [7] as well as the pre-determined growth on patterned silicon substrates [8]. The vertically aligned CNTs (VACNTs), uniformly grown by CVD on patterned surface features unique mechanical, electrical and thermal properties, that leads to propose a wide range of potential application of the system such as heat dissipation [9], biosensors [10], field emission sources [8], conductive electrodes and micro-mechanical devices [11].

An efficient and economical procedure of patterning the Silicon (Si) substrates by soft lithography, which allows growing VA-MWCNTs, is described in this paper. Taking advantage of this approach we have developed a number of CNTs based structures using precursors that allow for rapid growth and good CNTs quality. Furthermore, CNTs have been produced by the pyrolysis of cheap organic-metallic precursors which yields good quantity of MWCNTs under appropriate experimental conditions.

This work is also intended to show a specific application of such structures, namely forced air convective heat transfer enhancement on silicon substrate surface. Structures mimicking macroscopic finned heat sinks with dimensions of  $0.33\text{ mm} \times 0.47\text{ mm} \times 10.6\text{ mm}$  directly grown on silicon substrates of dimensions  $11\text{ mm} \times 11\text{ mm} \times 1\text{ mm}$  have been tested. Experiments were conducted within a wind tunnel where a convective heat transfer sensor, based on the notion of thermal guard, have been used [12, 13].

The key idea behind these tests is to show a possible application of MWCNT macroscopic structures in chip cooling. During last years the idea of using MWCNT-based structures for enhancing convective heat transfer has been largely explored. However, in most of those works, CNTs were intended to significantly enhance the heat transfer area. Therefore, the height of the MWCNT-based microstructures was chosen to be very small compared to thermal-fluid dynamic structures (i.e. thermal boundary layer thickness). Hence the enhancement in convective heat transfer was orders of magnitude smaller compared to the increase in heat transfer area.

For instance Fu et al. [14] built an on-chip ultra thin (50  $\mu\text{m}$ ) finned heat sink dissipator, whose heat exchange area is about 1000 times higher than traditional copper fins, which caused convective heat transfer augmentation of less than 15% using water as coolant, while no improvement was experienced when air was used as coolant. This probably happens because the thermal boundary layer in air is thicker than water (at same flow regimes), owing to a disparity in the Prandtl number. In [15], a MWCNTs coating on a surface is shown to cause an enhancement of 12% using air in natural convective heat transfer regime. In the work of Kordas et al. [16] microfins with base length of 50  $\mu\text{m}$  only produces a convective heat transfer enhancement of less than 15%.

Nevertheless, to the best of our knowledge, MWCNTs macroscopic structures in chip cooling application have not been sufficiently explored yet. Hence, section 4 of this work will be focused on this aspect.

## ***BUILDING OF MWCNTS STRUCTURES ON SILICON SUBSTRATE***

### ***Patterning by Photolithography***

We patterned Si wafer with Ultraviolet (UV) standard photolithographic technique in positive mode and image reversal mode, in order to obtain the growth of a well-defined structure of CNTs. Here we report some details about the whole procedure.

The Si (100) wafer cleaning procedure exploited, necessary in order to remove particulates on the wafer surface, as well as traces of organic, ionic and metallic impurities, includes the following steps (Figure 1-a): (i) ultrasonic bath in acetone for 10 minutes, (ii) soaking in 2-propanol for few minutes, (iii) bath for 5 minutes in Piranha solution ( $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$ , 3:1) and finally (iv) excessive bath rinsing in de-ionized water and drying with nitrogen gas. In order to remove residual moisture on the surface, after the cleaning procedure the Si substrate is placed for 2 minutes at 105 °C on a hot plate. Subsequently image reversal photoresist AZ5214E from MicroChemicals was spin coated on Si substrates using a spinner programmed at 600 rpm for 5 seconds and at 4000 rpm for the subsequent 40 seconds (Figure 1-b).

After uniform application of photoresist, Si substrate is soft baked at 120°C for 2 min (Figure 1-c). The photomask is then aligned with the wafer, so that the pattern can be transferred onto the wafer surface precisely. The resist-coated silicon wafer is brought into physical contact with the photomask (Contact Printing). The photoresist is exposed for 20 seconds with a Broad band UV source (Hg vapors lamp) with a power density of 3 mW/cm<sup>2</sup> (Figure1-d).

The next step is to dissolve the soluble photoresist areas in 1:1 solution of AZ Developer in deionized water and achieve a visible pattern on the wafer. Areas exposed to UV Light were removed after development, leaving bare windows on Si. In case of wafer with negative photomask (image reversal mode), few more steps are involved after UV exposure. i.e. reversal bake at 120°C for 2 min ( Figure 1-g) and flood UV exposure without mask for 70 seconds (Figure 1-h). The photoresist is directly developed in 1:4 solution of AZ 351B developer in deionized water.

After Si substrate patterning, the next step is to coat with a metal layer the areas where we don't want CNTs growth to occur. CNTs can be grown on a silicon substrate using a carbon source and the appropriate active metal catalyst. On other hand, non-catalytic metal can be used to restrain the growth of CNTs. So, we have used copper to cover the substrate where we do not want the growth of CNTs [17]. Metallic deposition is done by electron beam evaporation in a

vacuum chamber at a pressure of  $10^{-7}$  torr. At first, titanium (Ti) was deposited with a layer thickness of 20 nm and then 200 nm thick copper (Cu) was added on top of it (Figure 1-i). Titanium (Ti) is used as adhesion layer to overcome the poor adhesion of Cu to Si and to improve the stability of the deposited metal. Finally, “Lift-off” process has been carried out putting the Si substrate in an ultrasonic bath at 60°C for 15 minutes in NMP (N-Methyl-2-pyrrolidone) in order to remove the photoresist and metallic coating on the top of it and leave only the film which has been deposited directly on the Si substrate (Figure 1-j).

### ***Patterned growth of MWCNTs by Thermal CVD***

Growth of MWCNTs on patterned silicon wafers has been performed by thermal CVD of reagents consisting of commercially available carbon source and catalyst bought from Sigma-Aldrich. Ferrocene is used as a catalytic source because it is a good precursor to obtain iron nanoparticles, which leads to the formation of CNTs [17]. Camphor was preferred as carbon precursors because of its low cost and non-toxicity. In addition, the arrangement of pentagonal and hexagonal rings of carbon atoms in the camphor makes it a good source for the production of CNTs [18].

The CVD unit used for CNTs growth is shown in Figure 2. A 100 cm long horizontal steel tube was placed in a 3-Zone furnace which has the ability to maintain a uniform temperature up to 1200°C throughout its length i.e. 60 cm. Inside the tube, Si substrate is placed in the center of the oven in an inert atmosphere maintained by constant nitrogen/argon gas flow at 420 ml/min. The gas pressure is maintained slightly above atmospheric pressure to ensure laminar flow. A pyrex flask containing the reagent mixture of camphor and ferrocene (20:1) is connected to the steel tube with a T joint between the furnace and nitrogen inlet. The pyrex flask assembly was rested on a hot plate.

Furnace temperature is upheld at 850°C and in the meantime, pyrex flask is heated until evaporation of reagents which starts at about 200°C. The vaporized reactants were transported in

the oven with gas flow. The uniformity of the vapors flow was maintained by the temperature of the flask. The iron particles work as catalyst and the deposition of carbon species on Si substrate resulted in the growth of CNTs. After a suitable time i.e. 60 to 90 minutes, depending upon the required thickness of the CNTs carpet, the furnace was turned off. The method is described in further details elsewhere [19] and the growth parameters selection is described by S. Porro et. al [20].

## ***STRUCTURAL CHARACTERIZATION***

After growth of MWCNTs on the patterned surface, these structures were examined by field emission scanning electron microscopy (FESEM, Zeiss Supra 40). In Figure 3(a, b), the circular patterning of different sizes on silicon substrate is shown. Vertical columns of CNT grown by CVD shows a circular column width and length up to 1.5 millimeters and a diameter of 250 and 500 nm, respectively in Figure 3-c and Figure 3-d. The length of this column is usually in the range of several micrometers to a few millimeters.

Figure 4 shows some MWCNTs based structures grown in our CVD system, varying in the distance, size and the structure.

Figure 5 shows the structure, orientation, and size of CNTs in a certain structure. The diameters are mainly in the range of 30-70nm. Length of CNTs is up to several hundred micrometers and most of the CNTs are vertically well aligned.

CNTs have also been qualitatively and quantitatively characterized by Raman spectroscopy (Renishaw Ramascope Micro Raman) as shown in Figure 6-a and Energy Dispersive X-ray Spectroscopy (Figure 6-b) respectively to gain information about their structure. EDX spectra confirm the presence of around 2 wt% of catalyst particles of iron along with carbon material. Moreover Raman Spectra confirms the structure of CNTs as all the peaks relevant to MWCNTs are evident.



The Raman spectra of CNTs contain rich information about the electronic states and the phonon dispersion as it involves strong resonances of the incoming and outgoing photons and the vibrational states with the electronic energy levels of a tube. The D mode (Breathing Mode,  $A_{1g}$ -band) is peaked in the 1300-1400  $\text{cm}^{-1}$  range when excited with a visible laser. It is a typical grain size related disorder band observed in MWCNTs. The G mode (Tangential Mode,  $E_{2g}$ -band) is peaked in the 1550-1615  $\text{cm}^{-1}$  range and corresponds to the stretching mode of  $\text{sp}^2$  bonds in the graphite plane. A second-order-mode based on double resonance mechanism, often called G' mode is observed between 2620 and 2775  $\text{cm}^{-1}$ . Its intensity increases with the decrease in lattice disorder [21].

## ***CONVECTIVE HEAT TRANSFER CHARACTERIZATION OF MWCNTS STRUCTURES***

### ***Experimental Setup and Procedure***

To accurately measure the convective heat transfer, a sensor based on the notion of thermal guard is used in this work. This sensor (Figure 7) is made of:

- A copper cuboid of geometrical dimension 11 x 11 x 5 mm, named “core”, with an electrical heater placed beneath, while the specimen to be tested is glued on the core upper surface. A measurable heat flux crosses the core and the specimen, before reaching the coolant fluid.
- An isothermal element, namely the thermal guard surrounds all the facets of the core except the one that is in front of the air-flow. During the experiments, the latter guard is kept at the same temperature of the core itself by varying the power of a second dedicated heater.

The presence of the guard allow to minimize the spreading conductive heat losses, thus ensuring that all the thermal power generated by the core heater is directed to the flushing air. For additional information about the sensor design and applications to micro-structured surfaces, the reader can refer to Refs. [12, 13].

In [12, 13] no specimen was considered, hence the sensor surface exposed to the air flow appeared as a unique element. However, in this work, specimens consist of either silicon surfaces with MWCNTs structures (MWCNTSs finned surface) or the silicon surface alone (Figure 8). Specimens are attached on the core, so that a specimen is not aligned with the guard. As a result, on the wind channel wall, a protrusion with height equal to the specimen thickness is observed (roughly 1 mm). All specimens present a base surface area of  $1.23 \text{ cm}^2$  (square with approximately 11 mm edges).

The adopted sensor for the convective heat transfer coefficient is installed in the middle of the vertical wall of a horizontal wind channel, realizing a small open-loop wind tunnel. The channel is characterized by a smooth inner surface, a rectangular cross section of 228 mm x 158 mm (hydraulic diameter: 187 mm) and entrance length of 5 m (corresponding roughly to 26 hydraulic diameters).

Air is blown by a Savio s.r.l. centrifugal fan type SFL 25-A, with a throttling valve for regulating the mass flow rate. At the end of the channel, downstream with respect to the test section, a vane anemometer Testo 450 by Testo AG (accuracy  $\pm 0.1 \text{ m/s}$ ) was used for measuring the axial velocity.

Isothermal condition between core and guard is verified on the basis of both the upstream and downstream guard temperatures. Air temperature is measured at the same location of the anemometer. The channel wall temperature is monitored by a thermocouple installed on the inner surface of the channel. For all temperature measurements chromel-alumel (type K) thermocouples were used. Two HQ PS3003 variable power suppliers (voltage range 0-30 V and 0-3 A) are used to feed the sample heater and the guard heater, respectively. Minco electrical heater with a nominal resistance of  $26.5 \text{ }\Omega$  have been used. Thermal grease, with conductivity  $2.9 \text{ W/m/K}$ , was used for reducing thermal resistances at all contact surfaces of the device, when appropriate. Finally a six digits electronic multimeter (Agilent 34401A) is used to measure the voltage applied to the sample heater.

The average convective heat transfer coefficient  $h$  at the sample surface is computed as follows:

$$h = \frac{\frac{V_h^2}{R_h} - \sigma_B \varepsilon A (T_s^4 - T_w^4) - k \left\{ \frac{T_{g1} + T_{g2}}{2} \right\}}{A(T_s - T_a)} \quad (1)$$

where  $V_h$  is the electrical potential difference across the core heater resistance, whose value is  $R_h$ ,  $\sigma_B = 5.67 \times 10^{-8} \text{ W.m}^{-2}.\text{K}^{-4}$  is the Stefan-Boltzmann constant,  $\varepsilon$  is the average emissivity of the sample surface,  $T_s$  is the sample temperature measured by the thermocouple inserted in the center of the sample,  $T_w$  is the temperature of the channel wall,  $k=0.01 \text{ W.K}^{-1}$  is the sample-to-guard coupling thermal transmittance, which takes into account core-to-guard residual conductive parasite heat flux,  $T_{g1}$  and  $T_{g2}$  are the upstream and downstream temperatures in the thermal guard,  $A=1.23 \text{ cm}^2$  is the base specimen surface, and, finally,  $T_a$  is the air temperature.

An estimate of the average surface emissivity  $\varepsilon$  was obtained as follows. The sample temperature is first measured by a calibrated thermocouple. Next, the surface emissivity required in a thermographic camera (NEC TH9100 Series Infrared Thermal Imaging Camera) is tuned in order to match the above value of (independently) measured temperature. This procedure provided  $\varepsilon = 0.63$  and  $0.9$  for silicon surface and MWCNTSs finned surface, respectively. The experimental setup and procedure described above is the same used in [12, 13].

## ***RESULTS AND DISCUSSION***

In Eq. (1) the base specimens surface  $A=1.23 \text{ cm}^2$  is used instead of the actual heat exchange area because base surface reflects the size of the specimens.

Convective heat transfer coefficient was evaluated for different velocities, in order to explore the nature of the heat transfer phenomenon in a wide range of Reynolds numbers.

The Reynolds number  $Re_L$  is associated to the mean velocity within the wind tunnel  $u$ :

$$Re_L = \frac{uL}{\nu} \quad (2)$$

With  $\nu = 1.493 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$  being the kinematic viscosity of air and  $L = 20 \text{ mm}$  being the characteristic length of heat source (namely the guard). Conversely, the average convective heat transfer coefficient  $h$  was expressed in terms of the dimensionless group  $Nu/Pr^{1/3}$ , whereas:

$$Nu = h \frac{L}{k} \quad (3)$$

is the Nusselt number and

$$Pr = \frac{\nu}{\alpha} \quad (4)$$

is the Prandtl number with  $\alpha = 2.224 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$  being the thermal diffusivity of air.

Results for the two specimens of Figure 8 are shown in Figure 9. Uncertainty calculation have been performed taking into account both type A and type B uncertainty. A coverage factor of 2, which leads to a confidence interval of 95%, was adopted. The error bars in Figure 9 show a maximum and average uncertainty of  $\pm 6$ , 13% and  $\pm 5.16\%$  respectively. Details about uncertainty calculation are reported in [13]. MWCNTs finned surface shows a maximum and average enhance in convective heat transfer coefficient of 40% and 20% respectively compared to the silicon surface alone. It can be noticed that the enhancement in thermal performance is of the same order of the increase in heat transfer area (50%). The increase in area is hence much more efficiently exploited by the present solution then in cited papers [14-16].

## ***CONCLUSION AND FUTURE PERSPECTIVE***

An efficient protocol has been developed for patterning silicon substrates with different shapes and sizes of CNTs grown by thermal CVD technique. These vertically aligned MWCNTs have been tested for thermal dissipation system preparing MWCNT-based macroscopic heat fins aimed to enhance forced convective heat transfer on a silicon substrate. Convective heat transfer coefficient has been experimentally measured for silicon surfaces with and without MWCNTs grown on it. The MWCNTs enriched configuration shows an enhancement in convective heat transfer of 40% and 20%, as maximum and average value, respectively (compared to the surface

without fins). As MWCNTs macroscopic structures in chip cooling application have not been sufficiently explored in literature, these results might represent a first step towards chip cooling based on MWCNTs structures directly grown on the silicon substrate.

## ***ACKNOWLEDGMENT***

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## ***NOMENCLATURE***

$A$	Specimen base surface area ( $1.23 \text{ cm}^2$ )
$h$	Convective heat transfer coefficient
$k$	Sample-to-guard coupling thermal transmittance ( $0.01 \text{ W.K}^{-1}$ )
$L$	Characteristic length of heat source (20 mm)
$Nu$	Nusselt number
$Pr$	Prandtl number
$Re_L$	Reynolds number
$R_h$	Resistance of the core heater resistance
$T_a$	Air temperature
$T_{g1}$	Upstream temperature
$T_{g2}$	Downstream Temperature
$T_s$	The sample temperature measured
$T_w$	The temperature of the channel wall
$V_h$	Potential difference across the core heater resistance
$u$	Mean velocity within the wind tunnel
$\nu$	kinematic viscosity ( $1.493 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ )

## **GREEK SYMBOLS**

- $\alpha$  Thermal diffusivity of air ( $2.224 \times 10^{-5} \text{ m.s}^{-2}$ )
- $\sigma_B$  Stefan-Boltzmann constant ( $5.67 \times 10^{-8} \text{ W.m}^{-2}.\text{K}^{-4}$ )
- $\varepsilon$  is the average emissivity of the sample surface

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### ***List of Figure Captions***

Figure 1 Steps involved in transferring of mask to the substrate in image reversal mode a). Cleaning of wafer, b). Uniform application of photoresist by spin coating, c). Soft baking at 120°C, d). In contact mask alignment on photoresist surface, e). Controlled UV exposure, f). Reversal Bake at 120°C, g). Flood exposure, h). Development of structures, i). Metallic deposition by electron beam deposition, j). Lift off process.

Figure 2 Thermal Chemical Vapor Deposition Growth System

Figure 3 Patterned silicon substrates (a) & (b) and MWCNT columns (c) & (d)

Figure 4 CNTs based structures grown by CVD Technique

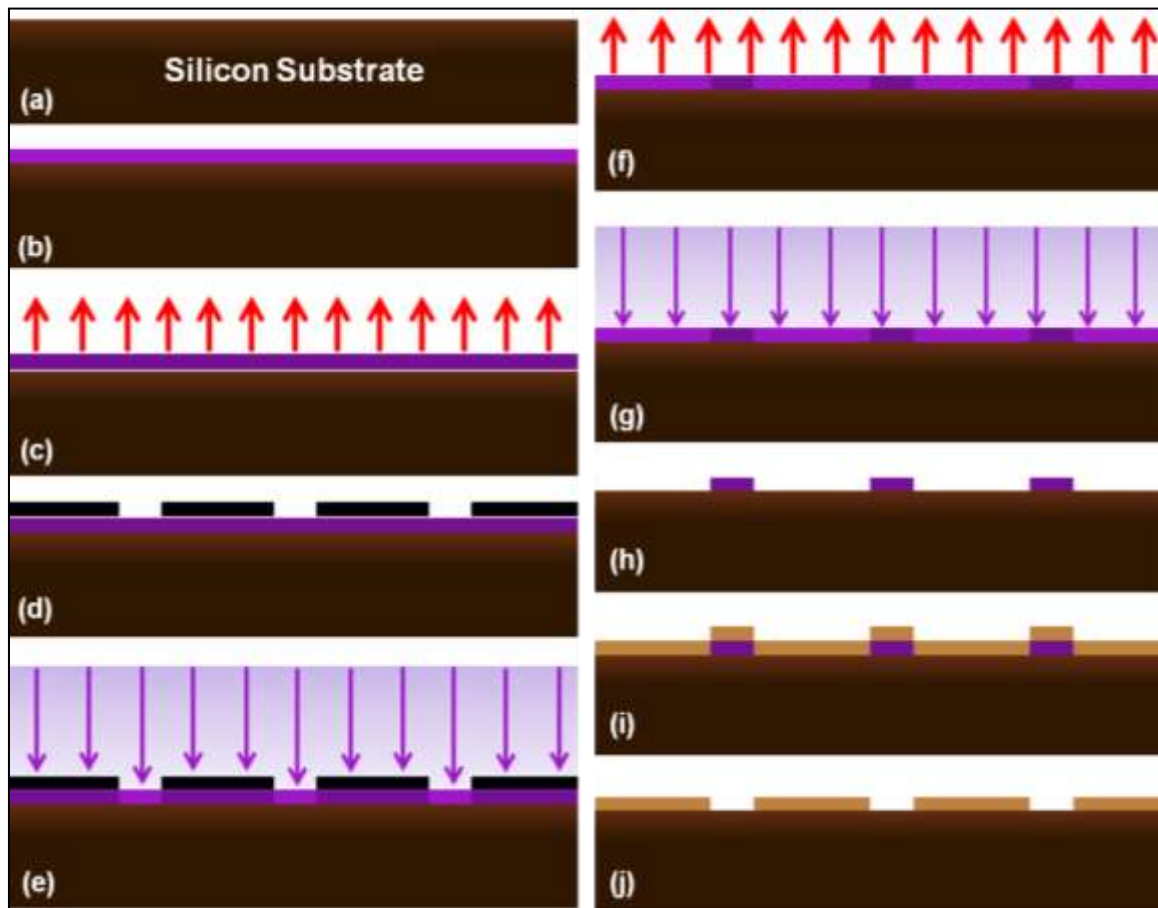
Figure 5 SEM micrographs of CNTs based structures

Figure 6 Raman Spectra (a) and EDX Spectra (b) of MWCNT based Structures

Figure 7 Specimens, (a) silicon surface, (b) MWCNTs finned surface. Fins are considered to be parallelepipeds of 10.6 mm length, 0.47 mm base and 0.33 mm height

Figure 8 Dimensionless heat transfer coefficient values in heat transfer coefficient of MWCNTs finned surface with regard to bare silicon one. All data are reported versus the Reynolds number. For MWCNTs finned surface and silicon surface (in Eq. (1)  $A = 1.123 \times 10^{-4} \text{ m}^2$  is the base area for all samples).

*Figures with Captions*



**Figure 9 Steps involved in transferring of mask to the substrate in image reversal mode a). Cleaning of wafer, b). Uniform application of photoresist by spin coating, c). Soft baking at 120°C, d). In contact mask alignment on photoresist surface, e). Controlled UV exposure, f). Reversal Bake at 120°C, g). Flood exposure, h). Development of structures, i). Metallic deposition by electron beam deposition, j). Lift off process.**

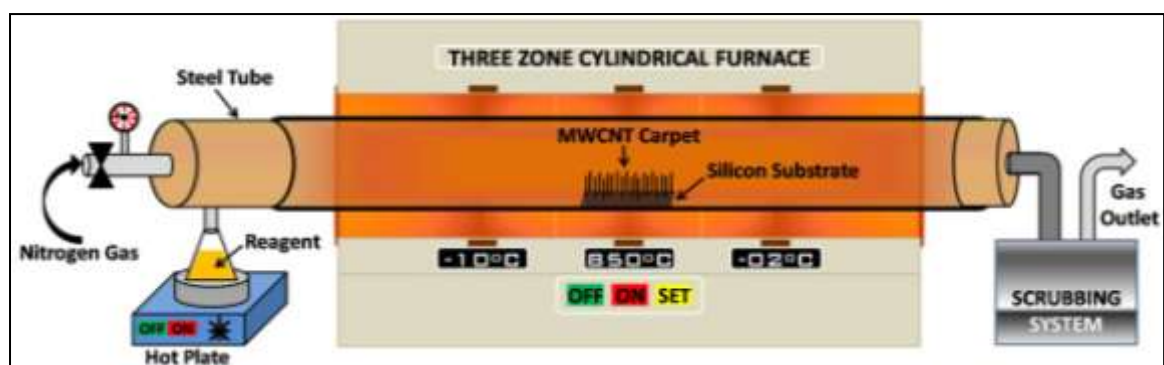
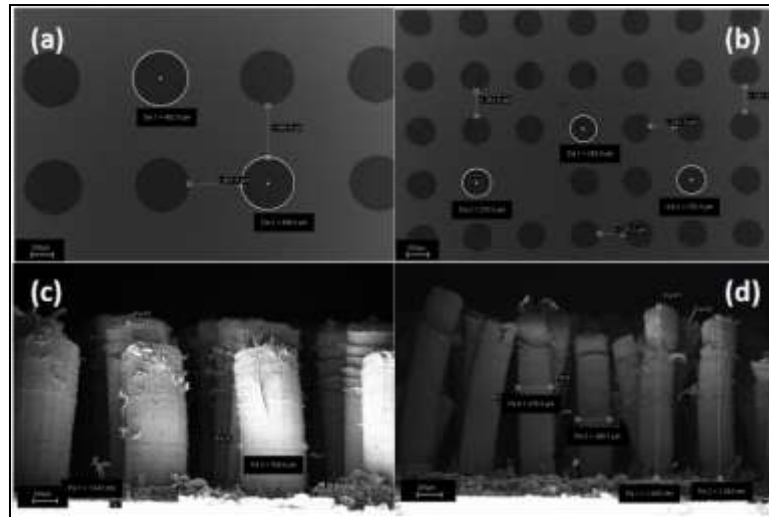
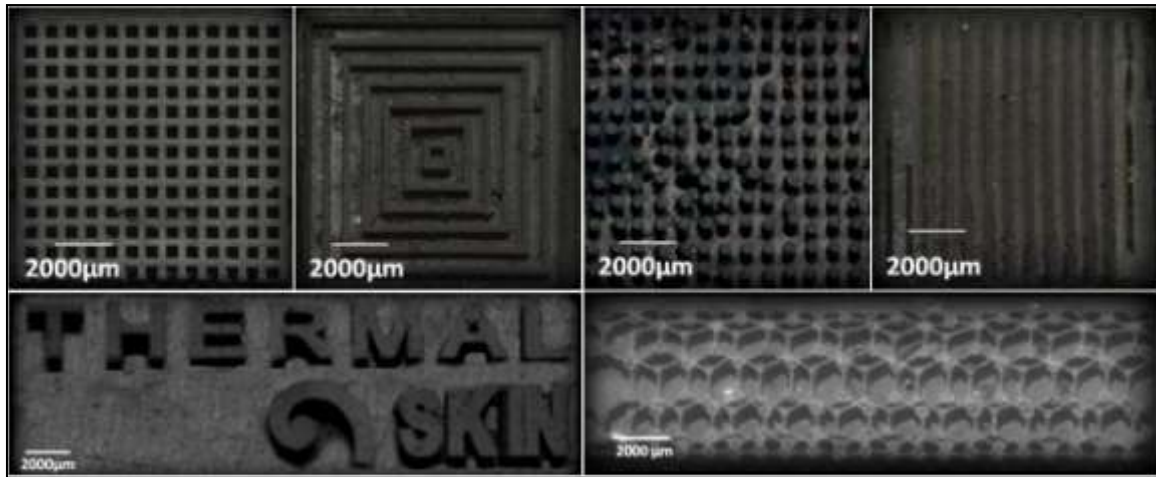


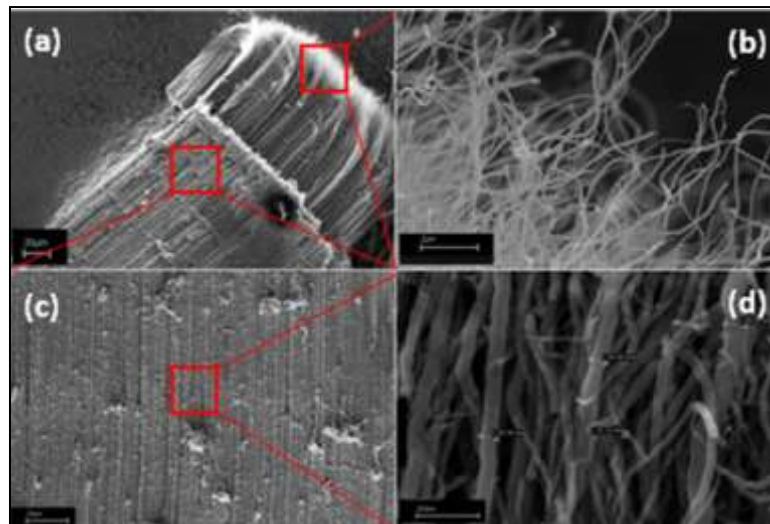
Figure 10 Thermal Chemical Vapor Deposition Growth System



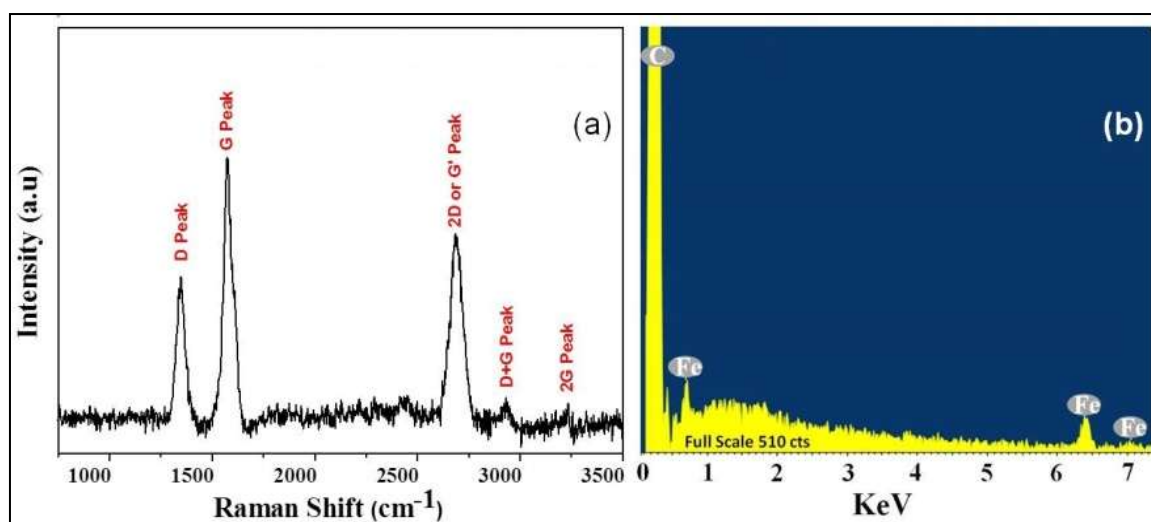
**Figure 11 Patterned silicon substrates (a) & (b) and MWCNT columns (c) & (d)**



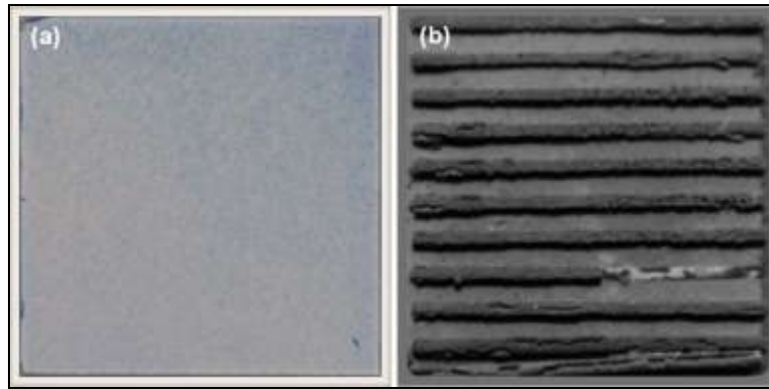
**Figure 12 CNTs based structures grown by CVD Technique**



**Figure 13 SEM micrographs of CNTs based structures**

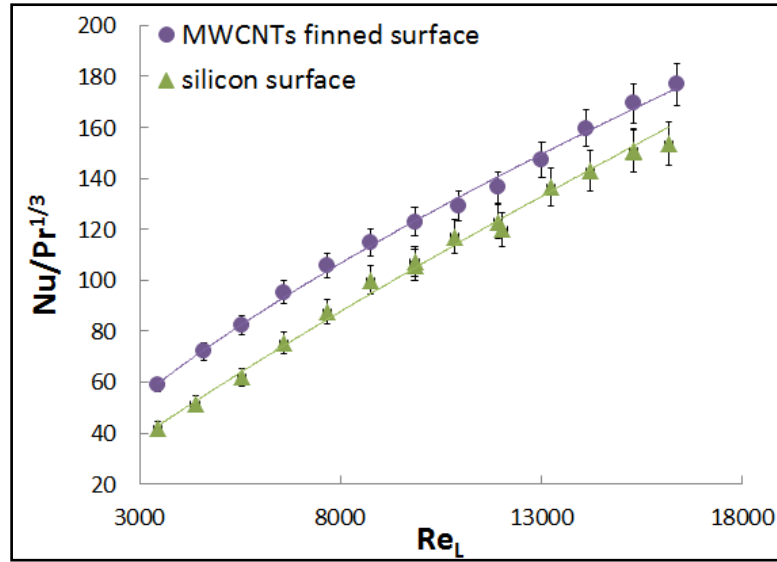


**Figure 14 Raman Spectra (a) and EDX Spectra (b) of MWCNT based Structures**



**Figure 15 Specimens, (a) silicon surface, (b) MWCNTs finned surface. Fins are considered to be parallelepipeds of 10.6 mm length, 0.47 mm base and 0.33 mm height**





**Figure 16 Dimensionless heat transfer coefficient values in heat transfer coefficient of MWCNTs finned surface with regard to bare silicon one. All data are reported versus the Reynolds number. For MWCNTs finned surface and silicon surface (in Eq. (1)  $A = 1.123 \times 10^{-4} \text{ m}^2$  is the base area for all samples).**



**Muhammad Imran Shahzad** was graduated in Applied Physics from University of Engineering & Technology-Lahore, Pakistan in 2009. He got his PhD degree in Physics from Department of Applied Science & Technology, Politecnico di Torino, Italy in 2014. Currently, he has been working as postdoctoral research fellow in Carbon Group at the Politecnico of Torino, Italy. The field of his research is about study of carbon based nanomaterials, their polymer composites and other potential application.



**Mauro Giorelli** received his B.S and M.S. in Electronic Engineering in 2004 and his Ph.D in Physics in 2008 from Politecnico di Torino (ITALY). Since 2005, he has (co-) authored over 30 publications with national and international partners. He is currently a post-doctoral researcher in Applied Science and Technology Department at Politecnico di Torino. He is a member of the Carbon Group, active in the field of carbon based nano materials and composites.



**Luigi Ventola** is a PhD Student. He received his B.S. and M.S. (cum laude) in Energetic and Nuclear Engineering in 2011. His primary research fields are thermo-fluid dynamics; granular flows and heat transfer.



**Denis Perrone** obtained his degree in Materials Science at the Università degli Studi of Turin, Italy in 2003, and then in 2007 his Ph.D. degree in Electronic Devices at Politecnico di Torino. He joined the Center for Space Human Robotics of Istituto Italiano di Tecnologia in Turin, Italy in 2011 as a technician. His skills are optical lithography, wet chemical etching, and PVD techniques for thin films coatings (thermal and electron beam-assisted evaporation and sputtering).



**Nadia Shahzad** is doing PhD in Physics at Department of Applied Science and Technology, Politecnico di Torino, Italy. She received her M.Sc. degree in Physics from University of Agriculture: Faisalabad, Pakistan in 2006 and then she completed her M. Phil in Applied Physics at the University of Engineering and Technology: Lahore, Pakistan in 2009. Her primary research fields are study of nanostructured thin films and their optical spectroscopic characterizations.



**Eliodoro Chiavazzo** received his B.S. and M.S. (cum laude) in 2003 and his Ph.D. in Mechanical Engineering in 2009 from the Swiss Federal Institute of Technology (ETH-Zurich). Since 2005, he has (co-) authored over 40 publications and 5 patents. In 2013, he has been awarded a Fulbright Fellow at Princeton University. His research fields are Systematic model reduction in multi-scale systems, Heat transfer enhancement and Heat transfer at micro- and nano-scale.



**Pietro Asinari** is an Associate Professor in Energy Department of Politecnico di Torino. He received his B.S. and M.S. (cum laude) in Mechanical Engineering in 2001 and his Ph.D. in Energetics in 2005. Since 2002, he has (co-) authored over 56 publications. In 2005, he won the ENI Award. His research fields are Transport theory at different scales, Kinetic modeling, Classical Molecular Dynamics, Thermodynamics, Numerical modelling & HPC and Heat Transfer.



**Matteo Cocuzza** graduated in Electronic Engineering at the Polytechnic of Turin in 1997. He received his PhD in Electronic Devices in 2003. Since 2004 he is a researcher of the C.N.R. Since 1998, he is involved in the development of MEMS for sensor applications and micro- and nano-platforms (Lab-on-a-chip) for genomic and proteomic applications. He is one of the founders of the Chilab - Materials and Microsystems Laboratory of Politecnico di Torino founded in 1998 and the Latemar National Laboratory, a Centre of Excellence funded by Miur in 2005.



**Alberto Tagliaferro** is associate Professor at Department of Applied Science & Technology Department of Politecnico di Torino. He got his degree in Nuclear Engineering and his PhD in Physics at Polytechnic of Turin. He is head of the Carbon Group, a research group founded in 2002. His research interest have moved in recent years from amorphous carbon and its alloys in thin film form to carbon nanotubes growth and applications (polymer filling, electrical applications, biosensors), and to color detection and reproduction. He is also associate editor of Diamond and Related Materials, an elsevier journal and chair of the thin film division of IUVESTA.

